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N'-Hydroxypyridine-2-carboximidamide

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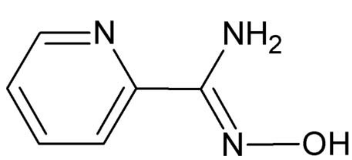
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.081; data-to-parameter ratio = 10.5.

The title molecule, $C_6H_7N_3O$, is almost planar (r.m.s. deviation = 0.0068 Å) and adopts an E conformation about the C=N double bond. In the crystal, molecules are linked by pairs of strong N—H···N hydrogen bonds, forming inversion dimers with $R_2^2(10)$ motifs. The dimers are further linked into $C(3)$ chains through O—H···N hydrogen bonds.

Related literature

For the pharmaceutical and biological activity of substituted N'-hydroxybenzamidines and 1,2,4-oxadiazole derivatives, see: Kundu *et al.* (2012); Sakamoto *et al.* (2007); Tyrkov & Sukhenko (2004). For a related structure, see: Sreenivasa *et al.* (2012)



Experimental

Crystal data

$C_6H_7N_3O$
M_r = 137.15
Monoclinic, $C2/c$
a = 21.367 (5) Å
b = 4.6382 (11) Å
c = 13.003 (3) Å
β = 105.468 (12)°
V = 1242.0 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.33 × 0.25 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.966, T_{max} = 0.979
8242 measured reflections
1086 independent reflections
982 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.081
S = 1.08
1086 reflections
103 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N3^{i}	0.903 (18)	1.859 (19)	2.7537 (14)	170.5 (17)
N2—H2N2···N1^{ii}	0.86 (1)	2.44 (1)	3.1753 (16)	144 (1)

Symmetry codes: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x, -y, -z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6916).

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supplementary materials

Acta Cryst. (2013). E**69**, o1180 [doi:10.1107/S1600536813017418]

N′-Hydroxypyridine-2-carboximidamide

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Comment

Substituted *N*′-hydroxybenzamidines are important intermediates obtained during the synthesis of pharmaceutically important 1,2,4-oxadiazole derivatives (Kundu *et al.*, 2012). 1,2,4-Oxadiazole derivatives are well known for their biological activities such as anti-HIV (Sakamoto *et al.*, 2007) and anti-microbial (Tyrkov *et al.*, 2004). In this view, we synthesized the title compound to study its crystal structure.

The title compound, (I), crystalizes with a single molecule in the asymmetric unit. This is in contrast to (*E*)-3-chloro-*N*′-hydroxybenzene-1-carboximidamide, (II), (Sreenivasa *et al.*, 2012) which crystalizes with two molecules in its asymmetric unit. Compound (I) adopts an *E* configuration across the C=N double bond, as the OH group and the benzene ring are on opposite sides of the double bond, while the H atom of the hydroxy group is directed away from the NH_2 group. This is similar as observed in (II). In the packing, the molecules are linked to one another through strong intermolecular N—H···N hydrogen bonds into $R_2^2(10)$ motifs forming inversion dimers. The dimers are further linked into C(3) chains through O—H···N hydrogen bonds.

Experimental

To a solution of 2-cyanopyridine (1 mmol) in ethanol was added triethyl amine (2.5 mmol) and hydroxyl amine hydrochloride, $NH_2OH.HCl$ (3.5 mmol). The reaction mixture was stirred at room temperature for 12hrs. (The reaction was monitored by TLC). The solvent was removed and the crude product was purified by column chromatography using hexane and ethyl acetate as the eluent.

Single crystals required for X-ray diffraction measurements were obtained from slow evaporation of the solution of the compound in a mixture of ethanol and dichloromethane (1:4).

Refinement

The hydrogen atoms attached to N and O were located in difference maps and refined isotropically. The remaining H atoms were positioned geometrically and refined using a riding model, with C–H = 0.93 Å with isotropic displacement parameters set to 1.2 times of the U_{eq} of the parent atom.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

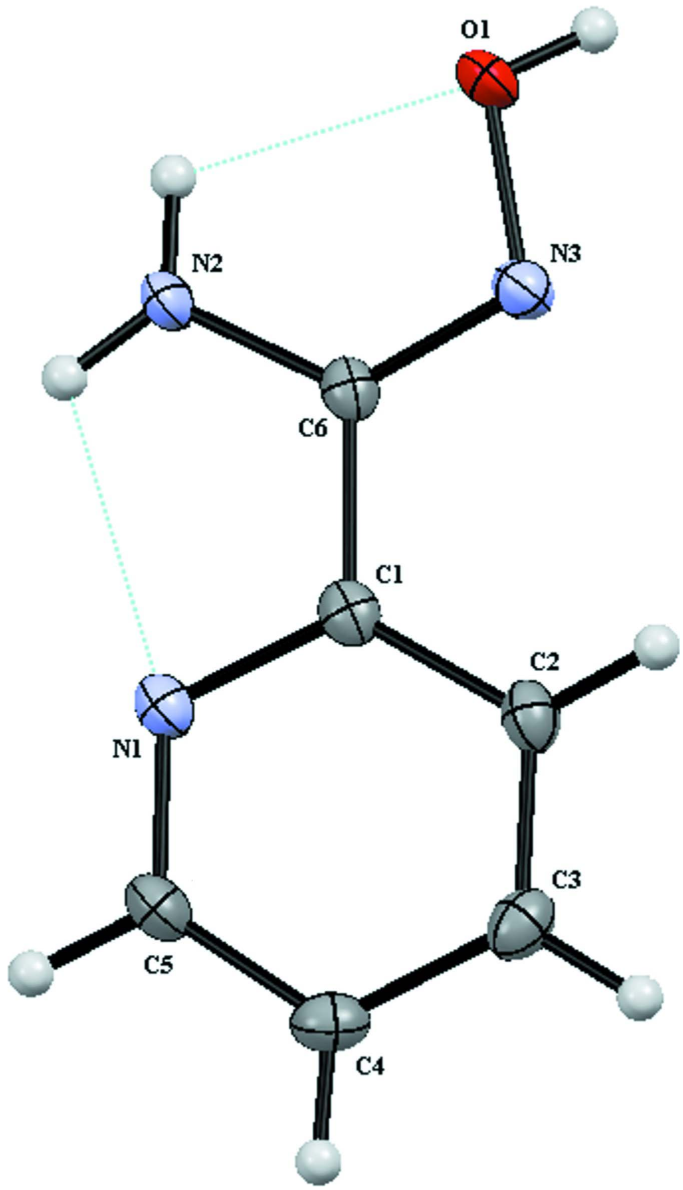

Figure 1

Molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

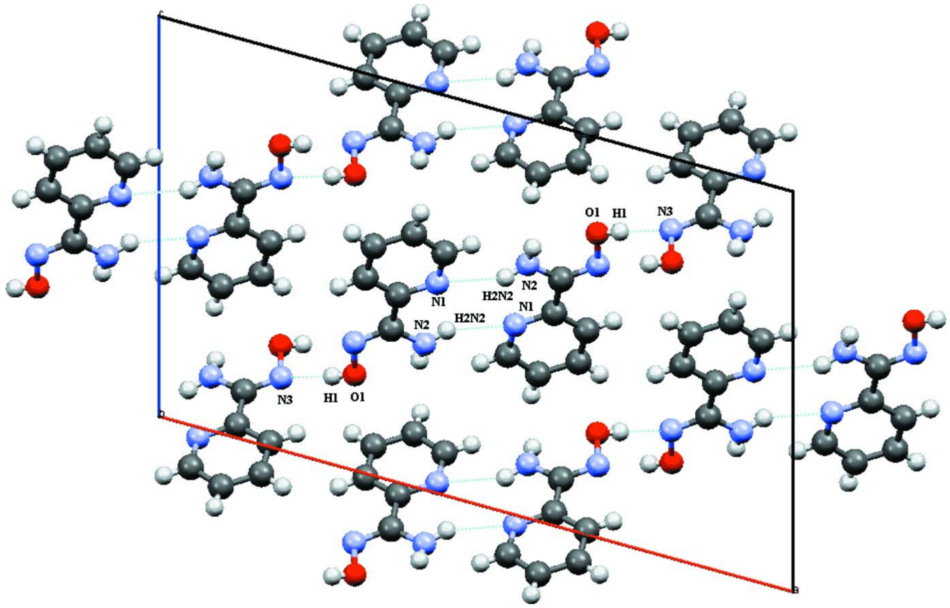

Figure 2

Packing of molecules displaying $R_2^2(10)$ loops and C(3) chains.

N′-Hydroxypyridine-2-carboximidamide

Crystal data

$C_6H_7N_3O$
M_r = 137.15
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 21.367 (5) Å
b = 4.6382 (11) Å
c = 13.003 (3) Å
β = 105.468 (12)°
V = 1242.0 (5) Å^3
Z = 8
F(000) = 576
prism
D_x = 1.467 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1088 reflections
θ = 2.0–25.0°
μ = 0.11 mm^{-1}
T = 293 K
Prism, colourless
0.33 × 0.25 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 1.03 pixels mm^{-1}
phi and *ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.966, T_{max} = 0.979
8242 measured reflections
1086 independent reflections
982 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −24→24
k = −5→5
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.081
S = 1.08
1086 reflections
103 parameters
2 restraints
0 constraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.8786P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.011$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.19134 (4)	0.1826 (2)	0.26040 (7)	0.0207 (3)
H1N2	0.0896 (7)	0.225 (3)	0.1797 (12)	0.027 (4)*
H2N2	0.0456 (6)	0.081 (3)	0.0861 (11)	0.027 (4)*
H1	0.2316 (9)	0.260 (4)	0.2779 (14)	0.043 (5)*
C1	0.12315 (6)	−0.2716 (3)	0.03188 (9)	0.0154 (3)
C2	0.17439 (6)	−0.4236 (3)	0.01036 (10)	0.0189 (3)
H2	0.2167	−0.3964	0.0520	0.023*
C3	0.16144 (6)	−0.6149 (3)	−0.07361 (10)	0.0208 (3)
H3	0.1949	−0.7164	−0.0904	0.025*
C4	0.09770 (6)	−0.6536 (3)	−0.13259 (10)	0.0211 (3)
H4	0.0874	−0.7834	−0.1891	0.025*
C5	0.05004 (6)	−0.4948 (3)	−0.10538 (10)	0.0203 (3)
H5	0.0073	−0.5229	−0.1449	0.024*
C6	0.13390 (5)	−0.0595 (3)	0.12070 (9)	0.0147 (3)
N1	0.06133 (5)	−0.3023 (2)	−0.02561 (8)	0.0176 (3)
N2	0.08163 (5)	0.0824 (2)	0.13540 (9)	0.0179 (3)
N3	0.19228 (5)	−0.0259 (2)	0.18081 (8)	0.0175 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0156 (5)	0.0240 (5)	0.0199 (5)	−0.0027 (4)	0.0004 (4)	−0.0070 (4)
C1	0.0157 (6)	0.0151 (6)	0.0149 (6)	−0.0009 (5)	0.0032 (5)	0.0048 (5)
C2	0.0152 (6)	0.0215 (7)	0.0186 (6)	0.0008 (5)	0.0022 (5)	0.0038 (5)
C3	0.0225 (7)	0.0210 (7)	0.0205 (7)	0.0050 (5)	0.0087 (5)	0.0035 (5)
C4	0.0276 (7)	0.0199 (7)	0.0157 (6)	0.0005 (5)	0.0055 (5)	−0.0008 (5)
C5	0.0175 (6)	0.0231 (7)	0.0173 (6)	−0.0017 (5)	−0.0006 (5)	−0.0008 (5)
C6	0.0142 (6)	0.0154 (6)	0.0141 (6)	0.0000 (5)	0.0029 (5)	0.0046 (5)
N1	0.0155 (5)	0.0193 (6)	0.0162 (5)	−0.0008 (4)	0.0012 (4)	0.0004 (4)
N2	0.0130 (6)	0.0216 (6)	0.0162 (6)	0.0013 (5)	−0.0009 (5)	−0.0031 (5)

N3	0.0163 (5)	0.0177 (6)	0.0169 (5)	−0.0009 (4)	0.0018 (4)	−0.0017 (4)

Geometric parameters (Å, º)

O1—N3	1.4201 (13)	C4—C5	1.3773 (18)
O1—H1	0.903 (18)	C4—H4	0.9300
C1—N1	1.3407 (16)	C5—N1	1.3408 (16)
C1—C2	1.3918 (17)	C5—H5	0.9300
C1—C6	1.4878 (17)	C6—N3	1.2928 (16)
C2—C3	1.3765 (18)	C6—N2	1.3532 (16)
C2—H2	0.9300	N2—H1N2	0.862 (13)
C3—C4	1.3849 (19)	N2—H2N2	0.859 (13)
C3—H3	0.9300		
N3—O1—H1	104.9 (11)	C3—C4—H4	120.9
N1—C1—C2	123.03 (11)	N1—C5—C4	124.19 (12)
N1—C1—C6	115.35 (10)	N1—C5—H5	117.9
C2—C1—C6	121.61 (11)	C4—C5—H5	117.9
C3—C2—C1	118.91 (12)	N3—C6—N2	123.75 (11)
C3—C2—H2	120.5	N3—C6—C1	118.26 (10)
C1—C2—H2	120.5	N2—C6—C1	117.97 (10)
C2—C3—C4	118.86 (12)	C1—N1—C5	116.69 (10)
C2—C3—H3	120.6	C6—N2—H1N2	116.3 (10)
C4—C3—H3	120.6	C6—N2—H2N2	119.9 (10)
C5—C4—C3	118.28 (12)	H1N2—N2—H2N2	118.9 (14)
C5—C4—H4	120.9	C6—N3—O1	108.89 (9)
N1—C1—C2—C3	−0.07 (18)	N1—C1—C6—N2	−0.29 (16)
C6—C1—C2—C3	−179.35 (11)	C2—C1—C6—N2	179.05 (11)
C1—C2—C3—C4	−1.19 (18)	C2—C1—N1—C5	1.51 (17)
C2—C3—C4—C5	0.96 (18)	C6—C1—N1—C5	−179.17 (10)
C3—C4—C5—N1	0.6 (2)	C4—C5—N1—C1	−1.77 (18)
N1—C1—C6—N3	178.22 (10)	N2—C6—N3—O1	−0.96 (16)
C2—C1—C6—N3	−2.45 (17)	C1—C6—N3—O1	−179.38 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3^{i}	0.903 (18)	1.859 (19)	2.7537 (14)	170.5 (17)
N2—H2*N*2···N1^{ii}	0.86 (1)	2.44 (1)	3.1753 (16)	144 (1)

Symmetry codes: (i) −*x*+1/2, *y*+1/2, −*z*+1/2; (ii) −*x*, −*y*, −*z*.